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(19)

(72) Inventors ALBERT EDWARD JOHN EVANS and PETER GATENBY TURNER

(54) HERMETICALLY SEALED FLEXIBLE PACKAGE

(71) We, KONINKLIJKE EMBALLAGE INDUSTRIE VAN LEER B.V., a Dutch corporate body of Amsterdamseweg 206, Amstelveen, the Netherlands, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to an hermetically sealed, flexible package.

Existing packages of this form are generally made from plies of metal, e.g. aluminium, foil laminated with a thermoplastic which makes it possible to form heat sealable joints between the plies. Although a sufficiently low permeability for a large number of applications can be obtained, nevertheless water vapour and other gases can permeate at a finite, albeit small, rate through the polymeric film which is exposed at the edges of the package, with no indication that this has occurred.

For some products even the small amounts of water or gas which can permeate into (or out of) the existing flexible packages may be unacceptable.

In accordance with the present invention there is provided an hermetically sealed flexible package comprising two superposed plies of material each including a pore free metal film coated on one surface, adjacent to and along the free edges thereof, with a solder, and a metal seal formed between the solder coatings by local application of heat and pressure, and sealing together the plies along the edges to form the closed package completely enclosing the package contents.

Such a package may be light-weight, easy with their solder coatings facing each other, applying heat and pressure to the edge areas of the superposed plies to melt the solder, thereby to form a metal seal between the solder coatings sealing together the plies, the heating being terminated before the pressure is released.

Packages according to the invention can be used to store materials which are very sensitive to influences of the environment, e.g. sterile products for medical and surgical applications, electronic components which are very sensitive to moisture and dust, perishable foods, air and/or water sensitive materials such as moisture or air curing adhesives and coatings. The packages can also be used to store sensitive articles which are to be kept in hostile environment, e.g. articles for aerospace applications.

The metal film of one or both plies may be supported on the side opposite the solder by a suitable heat-resistant plastics film, and may be coated partially or completely on the same side as the solder with a coating of a heat-sealable thermoplastic material. The thermoplastics coating may cover the solder in which case the metal seal is formed through the thermoplastics material by applying the heat to melt the latter so that the solder coatings are brought into direct contact for forming the seal.

The two plies may be separate and sealed together around their entire peripheries or alternatively may be provided by folding in half a single sheet of material in which case the three free edges of the plies will be sealed together. Under certain corrosive conditions, it may be that joint and seal integrity can only

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Existing packages of this form are generally made from plies of metal, e.g. aluminium, foil laminated with a thermoplastic which makes it possible to form heat sealable joints between the plies. Although a sufficiently low permeability for a large number of applications can be obtained, nevertheless water vapour and other gases can permeate at a finite, albeit small, rate through the polymeric film which is exposed at the edges of the package, with no indication that this has occurred.

For some products even the small amounts of water or gas which can permeate into (or out of) the existing flexible packages may be unacceptable.

In accordance with the present invention there is provided an hermetically sealed flexible package comprising two superposed plies of material each including a pore free metal film coated on one surface, adjacent to and along the free edges thereof, with a solder, and a metal seal formed between the solder coatings by local application of heat and pressure, and sealing together the plies along the edges to form the closed package completely enclosing the package contents.

Such a package may be light-weight, easy to open and suitable for radiation sterilization.

The invention also provides a method of manufacturing a flexible, hermetically sealed package system, comprising the steps of superposing two plies of material each including a pore-free metal film coated on one surface adjacent to and along the free edges thereof, with a solder, the plies being placed, together with their solder coatings facing each other, applying heat and pressure to the edge areas of the superposed plies to melt the solder, thereby to form a metal seal between the solder coatings sealing together the plies, the heating being terminated before the pressure is released.

Packages according to the invention can be used to store materials which are very sensitive to influences of the environment, e.g. sterile products for medical and surgical applications, electronic components which are very sensitive to moisture and dust, perishable foods, air and/or water sensitive materials such as moisture or air curing adhesives and coatings. The packages can also be used to store sensitive articles which are to be kept in hostile environment, e.g. articles for aerospace applications.

The metal film of one or both plies may be supported on the side opposite the solder by a suitable heat-resistant plastics film, and may be coated partially or completely on the same side as the solder with a coating of a heat-sealable thermoplastic material. The thermoplastics coating may cover the solder in which case the metal seal is formed through the thermoplastics material by applying the heat to melt the latter so that the solder coatings are brought into direct contact for forming the seal.

The two plies may be separate and sealed together around their entire peripheries or alternatively may be provided by folding in half a single sheet of material in which case the three free edges of the plies will be sealed together. Under certain corrosive conditions, it may be that joint and seal integrity can only be maintained by isolating the metal seal from the package contents and/or the external environment. Alternatively it may be desirable to prevent contamination of the contents of the package by components of the solder.

To meet these requirements a thermoplastic heat seal can be formed on one or both sides of the solder seal, if the films are provided

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with heat sealable thermoplastics coatings.

If required a desiccant, such as calcium chloride, lithium chloride or silica gel, and/or a gas or water sensitive indicator can be located within the package inside the solder seal. As used herein the term "solder" is intended to include tin-lead alloys and other metals and alloys which are heat fusible within the temperature limitations imposed by other components of the package. It is possible to form the solder seals without the use of a flux.

For ease of obtaining an adherent solder coating, copper foils are preferred but films of other metal or metal laminates may be used provided the solder can be applied to an adherent coating.

The published vapour permeability constants of polymers can be used to calculate the diffusion rates across polymer heat seals of known dimensions for defined conditions. The diffusion rate is directly proportional to the vapour pressure difference across the seal, inversely proportional to the length of the diffusion path, i.e. the width of the seal, and directly proportional to the cross sectional area of the diffusion path, i.e. the thickness of the seal multiplied by its average perimeter. Diffusion rates for a foil pack having a thermoplastic seal 2 mm wide, of total length 200 mm, and total thickness 0.05 mm, and for a gas or vapour pressure difference across the seal of 50 mm of mercury, were calculated from published permeability data and the results are given in Table 1.

TABLE I

Material	Gas	Quoted Permeability cc/cm²/sec/10 mm Hg	Calculated Diffusion rate: micro litres (at S.T.P.) per year	Calculated Diffusion rate: micro grams per year
Low density polythene 50 micron film	Water	2100×10^{9}	5500	4500
	CO_2	280	740	1450
	Oxygen	75	200	280
	Nitrogen	25	66	83
High density POLYTHENE 50 micron film	Water	300×10^{9}	790	640
	CO_2	65	170	340
	Oxygen	15	40	57
	Nitrogen	5	13	16
POLY PROPYLENE 50 micron film	Water	800×10^{9}	2100	1700
	CO_2	60	160	310
	Oxygen	20	50	75
	Nitrogen	4	10	13
SARAN (Registered Trade Mark) polyvinylidene chloride 50 micron film	Water	27×10^{9}	70	57
	CO_2	0.6	1.6	3.1
	Oxygen	0.05	0.1	0.2
	Nitrogen	0.01	0.03	0.03
POLY-VINYL CHLORIDE 50 micron film	Water	3200×10^{9}	8400	6800
	CO_2	2	5	10
	Oxygen	2.4	6	9
	Nitrogen	0.8	2	2.6

Some embodiments of the invention are described in detail below by way of example with reference to the accompanying drawing, in which:—

Figure 1 is an upper view of a package, having a solder seal and a single inner thermoplastic seal;

Figure 2 is a cross-section taken along the line X—Y of Figure 1;

Figure 3 is a detail cross-section through the seals;

Figure 4 shows an alternative form of seal; and

Figure 5 is a partial section through a pack-

age having thermoplastic seals either side of the solder seal.

The package shown in Figure 1 has an outer edge ABCD, and a solder seal EFGH extending around the edges of the package, a boundary KLMN being formed between solder in the edge area and a heat sealable plastics layer OPQR, and a space Z being formed for the material packed in the package. A composite seal S (Figure 2) of the package consists of the solder seal T and a plastics heat seal U, and seals together two plies around the space I for the packed material. Each of the plies comprises a metal foil III having a heat sealable plastics coating II. As shown in Figure 3 the metal foil III is supported by a heat resistant plastics film IV (Figure 3) and has a heat sealable thermoplastics coating IIB and solder coating IIA.

In Figure 3 the edge areas coated with solder are designated V and reference VI designates the edge area coated with the thermoplastics.

As shown in Figure 4 a pocket VII is formed between the plies and between two seals at the edge of the pocket, in which pocket a desiccant is located. In the package shown in part in Figure 5, a metal foil III of each ply has a supporting plastics film IV, a solder coating IIA, and a heat sealable thermoplastics coating IIB. Interconnecting the plies are a metal heat seal T and two plastics heat seals U. The solder seal extends over the width V while the plastic seals extend over portions VI. An unsealed outer edge area X is provided to facilitate peeling open the package.

The following examples elucidate the invention and also provide comparisons of the performance of joints made by conventional methods. In all examples, unless specifically stated otherwise, the methods of making the seals and testing the packages were the same.

Packages were made having seals made using a Sentinel (Registered Trade Mark) 12—12 AS laboratory impulse heat sealer using the impulse method of heating, i.e. the surfaces to be joined were compressed between two narrow tapes of resistance wire, a preset heating voltage was applied across the ends of the heating bands for a preset time, and after a further preset time to allow the molten joint to solidify the clamping pressure was released. The applied voltage and time taken were adjusted to suit the different material and solder coatings.

The effectiveness of the solder joints was established primarily by a helium leak test using a Veeco MS9 mass spectrometer. For these tests, helium was introduced into the packages immediately before the final edge was sealed. The test was carried out in an evacuated chamber. The apparatus was capable of detecting leak rates as low as 0.15 $\times 10^{-9}$ cc (at STP) per second. The test was repeated at least three times, thereby subjecting the test package to three evacuation cycles, which also provided an additional severe test of the mechanical integrity and strength of the seals.

A zero leak rate (i.e. $<10^{-10}$ cc He/sec) in the helium leak test was recorded and implied complete impermeability to water vapour. Nonetheless, packages were also tested for water impermeability. A water indicator comprising a label with a dried-on 10 microlitre drop of 10 milli-molar cobaltous chloride, dehydrated to the blue state, was sealed into each package. (This quantity of cobaltous chloride requires approximately 10 microgrammes of water to change from the anhydrous to the fully hydrated pink state.) Packages containing the water indicator were exposed to air saturated with water vapour at 79°C, i.e. to a water vapour pressure of 340 mm of mercury. This test, in addition to confirming the water impermeability of the joints, also shows the resistance of the packages to deterioration when exposed to extreme storage conditions.

The suitability of the packages for irradiation sterilisation was tested by exposing packages to a sufficient dose of gamma radiation to guarantee sterilisation, namely to 2.5 megarads. A radiation detection disc in the form of a Sessions Detex Radiation Indicator Label was included in each package to indicate whether an adequate irradiation dose had reached the contents. Results were found satisfactory.

Example 1.

Electroformed copper foil 33 microns thick, adhesively bonded to a supporting polyester film, sold by ICI under the Registered Trade Mark MELINEX, 25 microns thick, was electroplated with tin-lead solder, containing 60% by weight tin and 40% by weight lead, to a thickness of 3 microns. A thin coating of non-corrosive activated resin flux, marketed under the Trade Marks ALPHA 711—35 RELIAROS was applied. Square packages were first made from two 11 cm squares of this material by producing heat seals 1 cm in from the four sides using an impulse sealer as described above. The voltage was adjusted at 37.5 Volts, so as to produce a heat sealed joint in the tin-lead solder in 0.5 second. The square packages were cut in half, to make two open packages each 5.5 $\times$ 11 cm. Water and radiation indicators were placed in the packages which were then filled with helium before the open sides of the packages were sealed. The packages were tested as described, and the results are given in Table 2.

Example 2.

The copper-polyester film described in Example 1 was coated with a vinyl heat sealable lacquer (SWALE FT 3244) in a pattern

of rectangles corresponding to the areas within the soldered joints of the final packages described in Example 1. The uncoated areas were electroplated with 60—40 tin-lead solder to a thickness of 14 microns. No flux coating was applied. Pairs of packages were made from squares of this material as in Example 1, except that a second, plastics seal was made along each package edge, inside the solder seal, in the areas coated with the heat sealable lacquer. Thus the final packages had a complete double seal comprising an outer metallic or solder seal and an inner plastics seal produced between the lacquer coatings. These packages were filled and tested as described above and the results are also given in Table 2. The Voltage value used for producing the seals, metal and plastics, was 37.5 Volts.

Example 3.

Packages were made as described in Example 2, except that the solder coating was applied to a thickness of 8 microns and an additional coating of heat sealable lacquer was around the periphery of the 11 cm squares. Packages 11 cm square were made with a solder joint about 1 cm in from the edges, a plastics lacquer seal within the solder seal, and a third, outer seal between the lacquer coatings applied to the outer edges of the squares. The metal seal was thus protected on both sides by plastics seals. Indicators and helium gas were sealed into the packages for testing purposes as in the earlier examples.

In the sealing process the voltage employed in the impulse sealer was 37.5 Volts for the metal seal, 37.5 Volts for the inner plastics seal and 30 Volts for the outer plastics seal.

Example 4.

The same copper-polyester squares as mentioned in Example 1 were electroplated with 8 microns of pure tin and given a light application of flux solution as in Example 1. Packages 5.5 cm × 11 cm were made as described in Example 1 and tested. The results are given in Table 2.

In the sealing process the voltage used in the impulse sealer was 45 Volts.

Example 5.

Copper-polyester squares, as used in the previous examples, were electroplated with 13 microns of indium. A thin coating of flux (as in Example 1) was applied. Packages were made as described in Figure 1, but using a lower heating voltage of 32.5 V. The results of the tests are givevn in Table 2.

Example 6.

The same copper-polyester squares were "tinned", using a soldering iron on the copper surface where joints were to be made, with indium-tin eutectic alloy. A thin coating of flux (as in Example 1) was applied to the indium-tin surface, and packages made as described in Example 1 by making heat sealed joints in the indium-tin coating. These packages were tested in the same way as the previous examples.

In the sealing process the voltage was 25 V.

Example 7.

Aluminium foil 25 microns thick, adhesively bonded to MELINEX (Registered Trade Mark) 50 microns thick, was electroplated with 12 microns of tin-lead solder after first pre-treating the aluminium surface. The pre-treatment comprised a zincate dip treatment (Cannings Bondal Process) followed by a thin copper electroplating treatment. (Cannings is a Registered Trade Mark.) Packages were made with heat sealed joints in the solder in the same way as described in Example 1 and these packages were tested, the results being given in Table 2.

The voltage used in the impulse heat sealer was 37.5 V.

Example 8.

Steel foil (tinplate rolled to a thickness of 25 microns) was cleaned and electroplated with 12 microns of 60% by weight tin, 40% by weight lead, solder. Packages were made as in Example 1 with and without a thin coating of flux (as in Example 1) preapplied to the solder coating. The results of tests performed on the packages are given in Table 2.

The voltage used for the sealing was 50 V.

Example 9.

A strip of filter paper, approximately 1 cm × 6 cm, was saturated with a concentrated solution of cobaltous chloride which was then dried ond desiccated to the anhydrous blue state. This strip had the ability to absorb water and, by turning pink, to give an indication that water had been absorbed, absence of a colour change indicating that the relative humidity had been maintained below that of the equilibrium vapour pressure of the hydrated (pink) state of cobaltous chloride.

Two sets of 11 cm copper-polyester squares were coated over an inner area with vinyl heat seal lacquer (SWALE FT 3244) and the outer edges were electroplated with 12 microns of tin-lead solder. A desiccant strip was attached along one edge on the lacquer coating of some of the squares. Solder heat seals were formed along the four sides of two squares to form a package, helium gas, water radiation indicators being placed in the package before sealing the fourth side. Heat seals were made in the lacquer coated areas either side of the desiccant strip and in the same positions on similar packages without the desiccant strip. The total impermeability of the pack was established by a helium leak test under vacuum. The effectiveness of the

desiccant in preventing or delaying permeation of water vapour into the pack was tested by cutting off the totally impermeable seal along the side of the package including the strip, and on the corresponding sides of the packages without the desiccant strip, and exposing the package to water vapour in the same test as was used for the other examples. The result of the tests are given in Table 2.

The voltage used in sealing the packages was 37.5 V.

Example 10.

A commercial aluminium foil 25 microns thick, i.e. thick enough to be free from pores, and coated with a vinyl heat seal lacquer, was used to make packages with only plastics seals around their four sides. The method used was generally similar to that used for the metal joints in Example 1, but a lower voltage of 30 V was used for forming the heat seals. A water indicator and helium gas were sealed into the packages for testing purposes. These packages were prepared to give a comparison between packages in accordance with the invention and those previously proposed. The test results are given in Table 2.

Example 11.

Packages were made using the same 13 micron copper, 25 micron polyester as used in the earlier examples by heat sealing a film of an ionomer resin sold under the Registered Trade Mark Surlyn A by Du Point, 300 microns thick between the copper surfaces using the same processes as previously described. In the sealing process the voltage was 37.5 V. The thickness of the material allowed the dimensions of the resultant seals to be measured, from which a theoretical estimate of the diffusion rate into or out of the pack was calculated, as follows:

Quoted helium permeability of Surlyn A film 786 cc/645 cm^2 (100 sq.in)/24 hrs/25.4 microns (0.001 in)/atmosphere

Area of diffusion path = thickness of sealed joint multiplied by overall length of seal = 280 microns $\times$ 264 mm

Length of diffusion path = Width of resultant seal = 7.5 mm

Pressure of helium = not known, but estimated at about ½ atmosphere

Calculated helium diffusion rate of package = 18×10^{-8} cc He/sec.

A similar calculation for the water diffusion rate at the water vapour pressure of the test at 79°C gave a value of 6 micrograms per 24 hours. (Based on quoted water vapour permeability at 30°C.)

Helium leak tests and water vapour exposure tests were carried out as for the previous examples, and the results are given in Table 2.

TABLE 2

Results of Tests carried out on Examples

Example No.	Helium diffusion* rate cc/sec.	Water diffusion** Indicator changed	Helium diff. after water-test	Radiation" Sterilisation	Helium diffusion rate after radiation sterilisation
1	zero	nil for 28 days	zero	positive	zero
2	”	” ” ” ”	”	”	”
3	”	” ” ” ”	”	”	”
4	”	” ” ” ”	”	”	”
9	”	” ” ” ”	”	”	”
6	”	” ” ” ”	”	”	”
7	”	” ” ” ”	”	”	”
8 (fluxed and non-fluxed)	”	” ” ” ”	”	”	”
9 (without desiccant)	”	Some permeation after 20 days (metal point removed from one side)	Not measured	”	Not measured
(with desiacant)	”	Nil (metal point removed from one side)	Not measured	”	Not measured

TABLE 2 Continued)

Example No.	Helium diffusion* rate cc/sec,	Water diffusion** Indicator changed	Helium diff. after water-test	Radiation " Sterilisation	Helium diffusion rate after radiation sterilisation
10	.300–1500 × 10^{-9} (4 samples)	yes, after 8 days	Not measured	positive	Not measured
11	19 – 28 × 10^{-9} (4 samples)	yes, after 8 days	Not measured	,,	Not measured
(Calculated – 18 × 10^{9} from the data in example 9)					

* zero indicates less than detectable minimum of 0.15 × 10^{-9} cc/sec.

** Nil = cobaltous chloride spot remained blue;
yes = blue colour of spot lost.

" Positive – yellow indicator disc turned red

EXAMPLES – SUMMARY

Example No.	Substrate metal	Solder type	Application of solder	Plastics seal	Remarks
1	copper, 33μ	tin-lead, 3μ	electroplating	none	
2	copper, 33μ	tin-lead, 14μ	electroplating	one	
3	copper, 33μ	tin-lead, 8μ	electroplating	two	
4	copper, 33μ	tin,, 8μ	electroplating	none	
5	copper, 33μ	indium, 13μ	electroplating	none	
6	copper, 33μ	indium-tin	tinning (melting-on)	none	
7	aluminium 25μ	tin-lead, 12μ	electroplating	none	all with copper treatment
8	steel foil, 25μ	tin-lead 12μ	electroplating	none	
9	copper, 33μ	tin-lead, 12μ	electroplating	two	with desiccant
10	aluminium, 25μ	none	–	one	vinyl lacquer
11	copper, 33μ	none	–	one	Surlyn A

Example 12.

An electro-formed copper foil 33 microns thick, adhesively bonded to a supporting MELINEX (Registered Trade Mark) film 25 microns thick, was electroplated with tin-lead solder containing 60% by weight tin and 40% by weight lead, to a thickness of 12.5 microns. Pairs of prepared sheets of material were placed together with their solder surfaces in contact, and their edges were sealed using a bar sealer, i.e. a sealing device which is applied hot. In some cases a flux was introduced at the solder-solder interface while in others the surfaces were as prepared. Sealing bar temperatures were varied from below the melting point of the solder (from 190°C to 232°C) up to a temperature at which degradation of the backing polymer film occurred, and the clamping pressures were varied within the capacity of the machine (5 to 80 psi line pressure) and the sealing time was varied from one to 30 seconds.

Assesssment of joint effectiveness was made by hand peel testing and visual observation. No measureable peel strength was apparent for samples in which no melting of the solder

occurred, whereas the samples where melting was apparent, including those in which flux was used, had variable peel characteristics and inspection confirmed an intermittent nature of the joint formed. Comparison with samples produced as described in Example 2 but with the exclusion of the heat sealable lacquer showed a totally different character of the two joints. Joints sealed with an impulse sealer had relatively uniform peel characteristics and obvious joint continuity whereas joints sealed with a bar sealer all exhibited discontinuity. It can therefore be concluded that a true metallurgical joint is not formed by using a bar sealer and therefore metallic seals for an impermeable package cannot be formed, at least in times up to 30 seconds, by the use of bar sealing techniques.

WHAT WE CLAIM IS:—

1. An hermetically sealed flexible package comprising two superposed plies of material each including a pore free metal film coated on one surface, adjacent to and along the free edges thereof, with a solder, and a metal seal formed between the solder coatings by local application of heat and pressure, and sealing together the plies along the edges to form the closed package completely enclosing the package contents.

2. A package according to claim 1, wherein the metal film of at least one ply is supported by a film of heat resistant plastics material on the surface opposite said one surface thereof.

3. A package according to claim 1 or 2, wherein the metal film of each ply is coated partly or completely with heat sealable plastics material on said one surface thereof, and at least one plastics seal is formed between said coatings of the two superposed plies.

4. A package according to claim 1, 2 or 3, wherein the solder consists of tin, tin-lead, indium-tin or indium metal.

5. A package according to any one of claims 1 to 4, wherein a gas or water sensitive indicator, and/or a desiccant is located in the package within an area bounded by the metal seal.

6. A package according to claim 1, wherein the package is formed from two separate, identical plies of material each including a plastics film supporting the metal film on the surface opposite the solder coating and a coating of thermoplastics material on said one surface of the metal film over areas inside and outside the solder coating, a plastics heat seal being formed between the thermoplastics coatings of the two plies inside and outside of the metal seal.

7. A method of manufacturing a flexible, hermetically sealed package system, comprising the steps of superposing two plies of material each including a pore-free metal film coated on one surface, adjacent to and along the free edges thereof, with a solder, the plies being placed together with their solder coatings facing each other, applying heat and pressure to the edge areas of the superposed plies to melt the solder, thereby to form a metal seal between the solder coatings sealing together the plies, the heating being terminated before the pressure is released.

8. A method according to claim 7, wherein the solder consists of tin, tin-lead, indium-tin or indium metal.

9. A method according to claim 7 or 8, wherein a gas or water sensitive indicator, and/or a desiccant is located between the plies within the area bounded by the solder seal when completed.

10. A method according to claim 7, 8 or 9, wherein the metal film of each ply has a coating of thermoplastics material on the same surface as and covering the solder, the metal seal being formed by applying heat to melt the thermoplastics material at the location of the solder seal to allow the solder coatings to come into contact for forming the seal.

11. A method according to any one of claims 7 to 10, wherein the solder seal is formed without using a flux.

12. An hermetically sealed package substantially as herein described with reference to the accompanying drawings and/or as described in Examples 1 to 9.

13. A method of manufacturing an hermetically sealed package according to claim 7 substantially as herein described.

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1 475 080 COMPLETE SPECIFICATION
2 SHEETS *This drawing is a reproduction of the Original on a reduced scale.*
SHEET 1

fig. 1

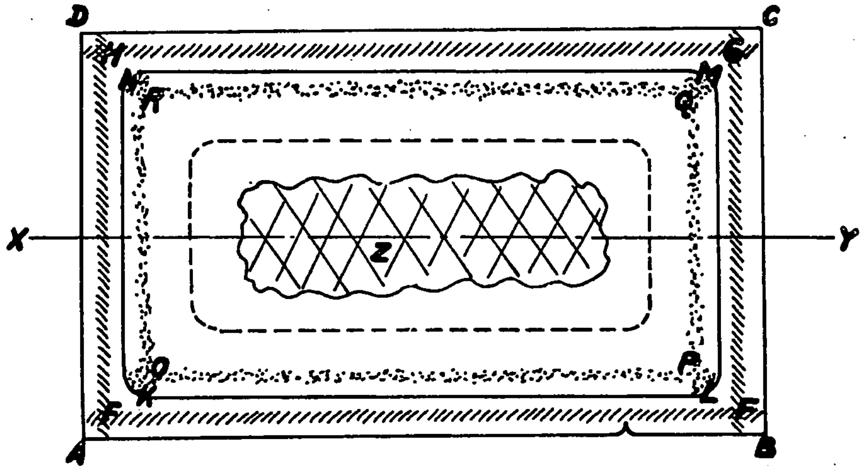


fig. 2

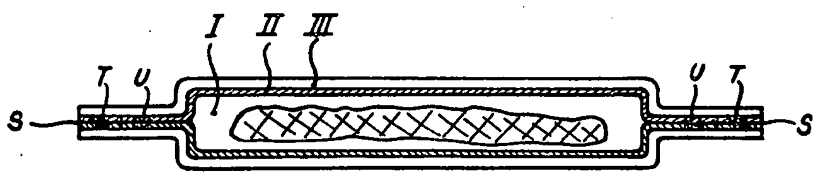


fig.3

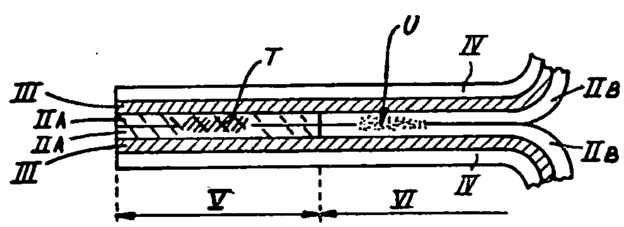
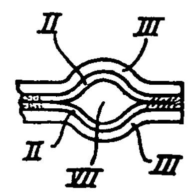


fig.4



COMPLETE SPECIFICATION

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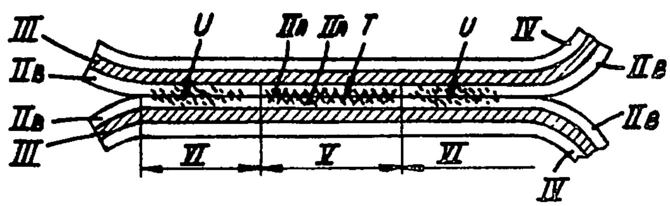
SHEET 2

fig.5



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